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BUREAU OF MINES

VAN. H. MANNING, DIRECTOR

A METHOD FOR MEASURING THE VISCOSITY OF BLAST-FURNACE SLAG AT HIGH TEMPERATURES

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A METHOD FOR MEASURING THE VISCOSITY OF BLAST-FURNACE SLAG AT HIGH TEMPERATURES.

By Alexander L. Feild.

INTRODUCTION.

The Bureau of Mines is investigating the problem of slag viscosity, its variation with the temperature and with the composition of the slag, and its effect upon the distribution of the sulphur between molten iron and slag. These investigations are being conducted in the laboratories of the Pittsburgh station of the Bureau of Mines, and will represent, when published, an introduction to a series of contributions to the theory of the metallurgical processes. This work is being carried on by F. H. Willcox, metallurgist in charge, and the author.

The relation of high temperature to the viscosity of blast-furnace slags, aside from being of considerable theoretical interest and value, has an important bearing on the metallurgy of iron. In this report is described a new method for determining the viscosity of blast-furnace slag at temperatures as high as 1,600° C. The convenient application of this method has necessitated the development of a suitable electric furnace, which is described in detail. A number of viscosity measurements on slags are given for the purpose of illustrating the general application of the method. The method and the furnace described were developed in the course of the investigations which are being conducted by Mr. Willcox and the author.

ACKNOWLEDGMENTS.

The author is indebted to J. K. Clement, physicist, for valuable help and information and for the use of apparatus and supplies. Acknowledgment is also made of the active interest and help of D. A. Lyon, metallurgist, under whose general supervision the work was undertaken. The chemical analyses of slags were made by F. D. Osgood, junior chemist, under the direction of A. C. Fieldner, chemist.

Acknowledgment is due W. F. Hausstein, instrument maker, and Walter Coons, machinist, for assistance in designing and making the furnace and apparatus.

THE LITERATURE ON SLAG VISCOSITY.

The viscosity of blast-furnace slag had not been measured previous to the investigation described in this report. A number of investigators have determined the "fusibility," or softening temperature, of slags by means of the cone method or other deformation method, but these methods supply only limited information regarding the temperature-viscosity relations of the slag in question. Any deformation method that may be employed indicates only the temperature at which the slag attains a more or less definite viscosity, the magnitude of the value obtained depending on the method used. The cone method was first carefully applied to the study of slags by Boudouard.[a]

Fulton [b] has determined the melting points of a series of copper slags by means of the thermal method of the Geophysical Laboratory. Such melting points correspond to that temperature at which the slag passes entirely from the crystalline to the amorphous state, and have no direct relation with the viscosity of the melt. Fulton obtained an idea of the temperature at which the slag became fluid by stirring the melt with a heavy platinum rod. The melting point temperature was much lower than the "fluidity" temperature, the difference between these two temperatures being extremely variable. An apparatus, consisting essentially of a graphite crucible with an elongated orifice in the base, has been devised by Johnson [c] for determining the "dripping point" of slags.

The application of customary methods of viscosity measurement have been very slowly applied to measurements at high temperatures, owing to experimental difficulties. In 1907 Lorenz and Kalmus [d] and Goodwin and Mailey [e] measured the viscosity of a number of molten salts of the alkali metals, lead, and silver by means of the capillary flow method up to temperatures of 500° to 600° C. In 1908 Fawsitt [f] employed the method of Coulomb [g] to determine the viscosity of mercury up to 216° C. and of sodium nitrate up to 450° C. His results for the sodium nitrate agreed with those of Goodwin and Mailey. In the same year Arndt [h] measured the vis-

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a Boudouard, O., Experiments on the fusibility of blast-furnace slags: Jour. Iron and Steel Inst., vol. 67, 1905, pp. 339–382.

b Fulton, C. H., The constitution and melting points of a series of copper slags: Trans. Am. Inst. Min. Eng., vol. 44, 1912, pp. 751–780.

c Johnson, W. McA., A new apparatus for determining the melting points of slags: Electrochem. and Met. Ind., vol. 4, 1906, p. 263.

d Lorenz, R., and Kalmus, H. T., Die Bestimmung der innern Reibung einiger geschmolzen Salze: Ztschr. Phys. Chem., Bd. 59, 1907, pp. 244–251.

e Goodwin, H. M., and Mailey, R. D., On the density, electrical conductivity, and viscosity of fused salts and their mixtures: Phys. Rev., vol. 25, 1907, p. 469; vol. 26, 1908, pp. 28–60.

f Fawsitt, C. E., On the determination of viscosity at high temperatures: Proc. Roy. Soc., vol. 80, 1908, p. 290.

g Coulomb, C. A.: Expériences destinées à déterminer la coherence des fluides et les lois de leur résistance dans les mouvements très lents: Mem. de l'Institut (Savants Etrangers), 1801, t. 3, p. 246; Recherches théoriques et experimentales sur la force torsion, et fur l'élasticité des fils de métal: Hist. de l'Acad., 1784, p. 229.

h Arndt, Kurt, Die Messung der Zahigkeit: Ztschr. Chem. Apparatenkunde, vol. 3, 1908, pp. 549–553.

cosity of boric anhydride, sodium metaphosphate, and mixtures of the two at temperatures of 700° to 1,100° C., by observing the time of fall of a platinum cylinder within the molten substance. Such a method is suitable for very viscous liquids only, and could be applied only with great difficulty to blast-furnace slags at working temperatures. Arndt also measured the viscosity of glass by this method at temperatures of 1,050° to 1,130° C. Doelter[a] in 1913 applied the method of Arndt to the determination of the viscosity of a synthetic diopside containing 5.02 per cent of ferrous oxide. A large temperature coefficient of viscosity was noted, the viscosity at 1,300° C. being five times that of castor oil. This isolated measurement by Doelter on a single mixture represents the highest temperature reached in the various viscosity investigations previous to those described in this report, in which results may be referred to absolute units. Arbitrary deformation tests have of course been made at much higher temperatures.

THEORY OF THE METHOD EMPLOYED.

In these investigations the author has used a modification of the method originated by Margules[b] in 1881. In Margules's method the liquid is confined between two concentric cylinders. The outer cylinder is rotated at a constant speed and the torque exerted upon the inner cylinder is measured. The method is applicable to liquids of a wide range of viscosity, and has been used, with modifications, by Gurney[c] in the measurement of the viscosity of water. The principle of this method is used in the Clark viscosimeter, which finds application in the physical testing of petroleum, oils, lubricants, and clay slips. According to Veinberg and Smirnov,[d] a method, similar in principle, except that the outer cylinder is stationary and the inner cylinder is rotated, is applicable to extremely viscous substances. The modification of the torsion method of Margules that has been used in the present investigation has been applied without difficulty to slags at temperatures as low as that corresponding to the softening temperature of the slag by the cone method, and, on the other hand, to slags at temperatures which correspond to working furnace temperatures—the temperatures covered corresponding approximately to a viscosity range of 200 to 3,000 (H_2O at 20° C. = 1).[e] The determination of slag viscosity over such a range of temperature was considered sufficient for the purposes of this investigation.

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[a] Doelter, C., Über die Viscositat der Silicatschmelzen: Chem. Ztg. vol. 36, 1912, p. 569.

[b] Margules, Max, Über die Bestimmung des Reibungs- und Gleitungs-coëfficienten aus ebenen Benegungen einer Flüssigkeit: Sitzungsber. Akad. Wiss., Wien. vol. 83, pt. 2, 1881, p. 588.

[c] Gurney, L. E., The viscosity of water at very low rates of shear: Phys. Rev., vol. 26, 1908, pp. 98–120.

[d] Veinberg, B. P., and Smirnov, I. A., Comparison of some methods of determining the viscosity of pitch: Jour. Russ. Phys. Chem. Soc., vol. 44, 1912, pp. 3–35; Chem. Abs., vol. 6, 1912, p. 3353.

[e] This unit of measurement, which is used for convenience, is equal to 1/100 C. G. S. unit.

Experimental data derived by means of the torsion method are capable of an easy, direct mathematical interpretation, in which respect it has a great advantage over other methods such as that of Segel,[a] in which the fall of a metal rod within a hollow cylinder is measured, the space between being filled with the viscous substance. The method of Arndt approaches more closely the conditions imposed by the use of Stokes's law governing the fall of a sphere in a viscous medium; but even here the interpretation of results is dependent on an assumption, which, however, is probably entirely permissible under the given experimental conditions.

In the torsion method the outer cylinder is rotated about a vertical axis at a constant speed. The inner cylinder is suspended coaxially within the outer one by means of a steel or phosphor bronze ribbon. The space between them is filled with the liquid, whose coefficient of viscosity may be designated as η. It may be shown[b] by means of the equation of Navier and the equation of continuity that a cylinder of radius b, rotating with a constant angular velocity, ω, will exert upon an inner fixed concentric cylinder of radius a (the space between them being filled with the liquid) a couple Γ, given by the relation

$$\Gamma = 4\pi\eta L \frac{a^2 b^2}{b^2 - a^2}\omega$$

where L is the common length of the two cylinders and the effects due to the ends is disregarded; and a and b are the radii of the inner and outer cylinders, respectively. It is at once evident that with cylinders of fixed dimensions the viscosity is proportional to the torsion couple Γ and inversely proportional to the speed of rotation in revolutions per second. Conversely, the torsion couple is proportional to the speed of rotation and also proportional to the viscosity. In the present experiments the observed end effect, or torsion couple arising from the shear exerted by the end surfaces, amounted to 23 per cent of the total effect. This end effect was determined by the use of two inner cylinders of equal diameter but of different lengths (5.08 and 2.70 centimeters). An appreciable fraction of the end effect was obviously caused by the immersed part of the cylindrical shaft supporting the inner cylinder. On account of the experimental conditions at high temperatures it was not possible to use a supporting shaft of very small diameter, such as is easily possible at lower temperatures. As is shown later, the torsion couple, inclusive of the end effect, is proportional to the speed of rotation and also to the viscosity of the liquid over a considerable range of speed and viscosity; so that the error arising from the end effect was probably within other known experimental errors. Hence it was not necessary

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[a] Segel, M. Über eine Methode zur Bestimmung der inneren Reibung fester Körper, Physikalische Ztschr., vol. 4, 1903, p. 493.

[b] See Gurney, L. E., The viscosity of water at very low rates of shear: Phys. Rev., vol. 26, 1908, pp. 98–120.

to apply any correction for variations of end effect for different viscosities of the liquid slag.

The theoretical conditions are, among other things, that the cylinders are concentric and that the conditions about the ends—that is, the depth of slag and relative position of surfaces—is the same in every case. Couette[a] in early experiments with this method has shown that in this case, as in others where the magnitude of a physical effect depends on the relative position of nearly concentric cylinders, a slight eccentricity produces an error relatively much smaller. Gurney[b] found that in the case of measurements with water as the liquid and with an inner cylinder of 4.5 centimeters radius, a displacement of 1 millimeter from the central axis of the outer cylinder caused an error which was inappreciable. He remarks: "Any departure from a truly circular cross section in the cylinders, or any tilting or lack of concentricity would cause an apparent increase in η, since the ideal case is the case of minimum deflection. The actual case may also differ slightly from the ideal in other ways, and the tendency would usually be to increase the apparent value of η." In the experiments described herein it is probable that the eccentricity of the inner cylinder did not exceed 1 millimeter, although an eccentricity of this magnitude was sometimes noted, without any appreciable effect on the observed deflection.

PROBABLE EFFECT OF SURFACE SLIP.

Twenty-three years after the pioneer work of Poiseuille (1842), in establishing the laws of internal friction in fluids, Helmholtz and Piotrowski[c] concluded from certain experiments, in which a sphere full of a liquid was allowed to oscillate, that there existed at least in some cases a "slip" at the boundary plane between solid and liquid. Whetham,[d] however, 25 years later, obtained convincing evidence from experiments with a silvered capillary that there was no "slip" for such liquids as were used by Helmholtz and Piotrowski. He attributed their results to an insufficient recognition of the effects of temperature and to an inconstancy in the bifilar suspension used. Whetham's conclusions have found substantial support from the later researches of Mutzel[e] (1891), Drew[f] (1901), Arnold[g] (1911), and Zemplen and Pogany[h] (1916). In general it seems that the more sensi-

[a] Couette, M., Études sur le frottement des liquides: Ann. chim. phys., ser. 6, t. 21, 1890, p. 433–510.

[b] Gurney, L. E., Op. cit., p. 110.

[c] Helmholtz, H., and Piotrowski, —, Sitzungsber. Akad. Wiss., Wien, vol. 50, 1865, p. 107.

[d] Whetham, W. C. D., On the alleged slipping at the boundary of a liquid in motion: Philos. Trans., vol. 181, 1890, p. 559–582.

[e] Mutzel, K., Ueber innere Reibung von Flussigkeiten: Wied. Annalen, vol. 43, 1891, p. 15.

[f] Drew, E. R., A determination of the viscosity of water: Phys. Rev., vol. 12, 1901, pp. 114–120.

[g] Arnold, H. D., Limitations imposed by the slip and inertia terms upon Stokes's law for the motion of spheres through liquids: Philos. Mag., vol. 22, pp. 755–775; Chicago University Theses, vol. 3, 20 pp.

[h] Zemplen, Gyozo, and Pogany, Bela, Untersuchungen uber die innere Reibung von Flussigkeiten Innere Reibung und Gleitung tropfbarer Flussigkeiten: Ann. der Physik., vol. 49, 1916, pp. 39–70.

tive the experimental method the smaller the possible value of the "slip" coefficient becomes.

Margules[a] has derived an expression for the torsion couple exerted upon the inner cylinder which takes into account the existence of a surface "slip." It is:

$$\Gamma=4\pi\eta L\frac{\omega}{2\gamma\left(\frac{1}{a^3}+\frac{1}{b^3}\right)+\left(\frac{1}{a^2}-\frac{1}{b^2}\right)}$$

where γ is the "slip" coefficient or "Gleitungskoefficient." When γ is equal to zero, the expression reduces to that previously given,

$$\Gamma=4\pi\eta L\frac{a^2b^2}{b^2-a^2}\omega$$

derived on the assumption that there is no slip between the solid and the liquid at the boundary plane.

In the present investigations the magnitude of the slip has not been determined, but has been assumed to be equal to zero in the case of the low rates of shear employed—about 1 revolution per second.

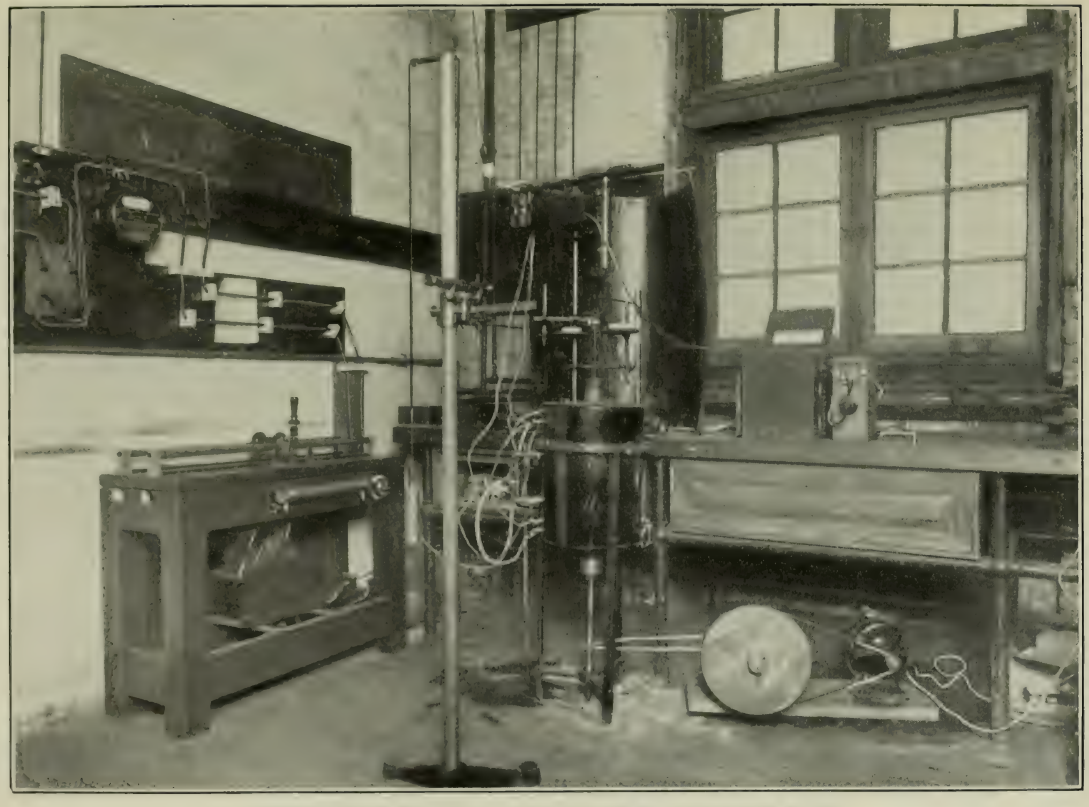
DESCRIPTION OF FURNACE AND ACCESSORIES.

The electric furnace used in the experiments of the author is of the granular carbon, resistance type, and is similar as regards the arrangement of resistor and supporting refractories to the one described by Jeffries.[b] The arrangement of the furnace and its accessories are shown in Plate I.

The heating zone is comparatively large—3 inches in diameter and 6 inches long—and consequently required very efficient heat insulation of the cylindrical part of the heating zone and also of the circular ends. The maintaining of high temperatures for a comparatively long period necessitated making the furnace air-tight by means of an external sheet-iron shell to protect the resistor from oxidation and prevent excessive generation of carbon monoxide. Exclusion of the atmosphere from the interior of the furnace was also necessary to protect the graphite crucible and viscosity spindle. In order to afford an easy means of renewing the granular resistor, inspecting the interior of the furnace, and introducing the graphite crucible containing the slag sample, the sheet-iron shell is divided horizontally along a plane which cuts the top of the heating zone. When the furnace is in use the upper part, which is removable, is bolted to the stationary lower part along machined flanges provided with asbestos gaskets. The outer furnace shell is 18 inches long, over all, and 14 inches in diameter. The heating zone occupies a central position within the shell. Electrical connection is made with

[a] Margules, M., Loc. cit.

[b] Jeffries, Zay, Notes on the gran-annular electric furnace: Met. and Chem. Eng., vol. 12, 1914, pp. 154–157.



APPARATUS FOR MEASURING VISCOSITY OF BLAST-FURNACE SLAGS.

graphite ring electrodes by means of graphite rods inclosed in a sheet-iron sheath. The graphite core of these electrode connectors is threaded into the electrodes proper. The connectors are continued through the sheet-iron shell and connected externally with the leads from the transformer, being insulated from the shell by means of asbestos packing. The outer ends of the connectors are water-cooled to prevent heating troubles.

The resistor material used in these experiments was granular carbon, although "cryptol" or graphite could be used as well. The granular carbon as obtained is screened to approximately 20-mesh size.

The inner and outer refractory tubes which inclose the resistor material are of Norton alundum. Both glazed and unglazed tubes have been used without any particular difference in their behavior being noted. The inner and outer tubes must be renewed at intervals, owing to cracking which apparently can not be avoided even with slow rates of heating and cooling. The life of the alundum is considerably prolonged if the furnace is not heated to temperatures above 1,500° C. The alundum heating tubes are immediately surrounded by magnesite chips, which in turn are insulated by kieselguhr from the outer shell. The end parts of the furnace are insulated with kieselguhr, which is protected where exposed to the intense heat of the heating zone by a layer of Northrup cement placed over 20-mesh magnesite.

As the upper part of the furnace was made removable, it became necessary to find a means of supporting the kieselguhr without introducing any fusible or heat conducting material. Asbestos and transite[a] board were tried, but immediately deteriorated because of dehydration and elimination of the binding material. Accordingly, fire-clay cylinders were placed both above and below the heating element, and the upper fire-clay cylinder was bolted to the top of the upper removable cylinder by means of lag screws. The kieselguhr packed between the fire-clay ring and the outer shell was prevented from falling out by covering the surface with a layer of Northrup cement.

The furnace is supported vertically by a tripod bolted to the sheet-iron shell, the bottom of the furnace being 22 inches above the floor of the room. A support is bolted to the upper, removable shell to which the optical pyrometer is attached when the furnace is in use.

ROTATING DEVICE.

A ¾-inch steel shaft, by means of which the cylindrical crucible containing the slag is rotated, projects up into the furnace through a stuffing box placed centrally in the base of the sheet-iron shell. The

[a] Commercial product, consists principally of asbestos, mixed with a refractory binding material.

lower end of the shaft is supported on a bearing fixed to the tripod. The steel shaft extends only a short distance beyond the bottom of the furnace shell, and is bored for the reception of a slightly smaller Marquardt porcelain tube, which extends up to the beginning of the heating zone. The graphite crucible, the construction of which is described later, is set into the porcelain tube. As the steel shaft extends 12 inches above the stuffing-box bearing, careful machining and adjustment is necessary in order to prevent any eccentricity in the rotation of the crucible. The use of the porcelain tube between the highly heated crucible and the steel shaft was considered necessary in order to prevent an undesirable conduction of heat from the slag within the crucible.

SOURCE OF HEAT.

The annular ring of granular carbon resistor is one-half inch in thickness. The heating current is supplied by a transformer equipped with an adjustable panel which gives voltages ranging from 40 to 100 volts in steps of 2 volts. A fine adjustment is obtained by a slide-wire resistance coil placed in the primary circuit. The maximum power input amounts to 5 kilovolt amperes and the maximum current to 110 amperes.

FURNACE ATMOSPHERE.

The normal atmosphere of the furnace is a mixture of carbon monoxide and nitrogen, and hence corresponds to that in the hearth of a blast furnace. During the initial runs with the furnace considerable quantities of combustible gas were generated. A sample of this gas was analyzed by G. A. Burrell, chemist of the bureau, and showed the following composition: Hydrogen, 47.46 per cent; carbon monoxide, 35.78 per cent; carbon dioxide, 9.75 per cent; nitrogen, 5.37 per cent; oxygen, 1.56 per cent. The gas was evidently formed by the action of water vapor, expelled from the kieselguhr, on the incandescent carbon resistor. This excessive generation of hydrogen and carbon monoxide diminished rapidly, owing to the expulsion of the water of combination of the kieselguhr as well as the moisture contained therein.

THE VISCOSITY APPARATUS.

Acheson graphite seems to be the only refractory available for such use as that of high-temperature viscosity measurements on blast-furnace slag. An apparatus or crucible made of platinum would be rapidly destroyed in such a furnace atmosphere, especially as all slags contain considerable quantities of iron and sulphur. Of the various refractory oxides generally used zirconia, the refractory

properties of which are described by Meyer,[a] comes nearest to fulfilling the necessary conditions, because it is not improbable that a refractory mixture may be made from it that will not flux appreciably with the molten slag. Such a refractory, however, was not available when these experiments were performed.

Acheson graphite possesses the advantages of having a very low coefficient of expansion, a relatively great mechanical strength at high temperatures, desirable working properties, and shows inappreciable alteration on being kept for long periods at high temperature. All of these properties are desirable for the construction of an apparatus which must of necessity preserve its shape and dimensions over a wide range of temperature. The action of the molten slag on the viscosity spindle immersed in it is so small that the radius of the spindle decreases only approximately 0.005 centimeter during a viscosity determination. The action of the slag is perfectly uniform over the surface of the spindle, so that the same spindle may be used repeatedly, due allowance being made for the slight change in its radius.

The construction of the viscosity spindle and connecting rod is shown in figure 1. On account of the difficulty of machining a graphite rod of any considerable length, possessing as small a diameter as is necessary in the construction of this apparatus, the viscosity spindle and connecting rod are turned separately in the lathe. When in use the top of the spindle is threaded into the base of the connecting rod. The construction permits easy renewal of the spindle in case of accidental breakage, which is liable to occur when the apparatus is handled without proper care. The upper part of the graphite connecting rod, when in use, is sufficiently cool to permit the use of a steel connecting collar for attaching the lower graphite part of the apparatus to the upper metal part of the suspension system, described below. The construction of the graphite crucible is shown in figure 2.

In determining the viscosity of ordinary liquids at room temperatures by the torsion method there is no particular difficulty in accurately adjusting the inner and outer cylinders and in maintaining a comparatively large volume of the liquid at the required temperature. Moreover the liquids are usually of such low specific gravity that the weight of the inner cylinder is in itself sufficiently great to maintain the suspended system centrally within the rotating outer cylinder. For these reasons the inner cylinder may be made comparatively large. There is also no difficulty under such conditions of extending the suspension wire or ribbon beneath the surface of the liquid.

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[a] Meyer, H. C., "Zirconia," a new refractory, Met. and Chem. Eng., vol. 5, 1914, p. 791; Further notes on the refractory properties of zirconia, vol. 7, 1915, p. 263.

At very high temperatures, however, such as are necessary in the determination of slag viscosity, the specific gravity of the slag is greater than that of the immersed graphite spindle; the volume of liquid slag that can be maintained at a uniform temperature is comparatively small; and from the nature of the case the steel suspension ribbon and mirror must be a considerable distance from the surface of the molten slag. In the furnace used the distance from the surface of the slag to the bottom of the steel suspension ribbon is approximately 21 inches; the spindle itself is only 1 inch long, and the steel ribbon about 1¼ inches long. Such a system would not in itself be at all stable under the conditions of measurement. It was therefore necessary to place a weight of approximately 200 grams between the suspension ribbon and the spindle at a point just outside the furnace shell and about 4½ inches below the suspension. This weight gives stability to the system, decreases the effect of vibrations upon the mirror, and holds the spindle in its position in the rotating slag mass. Moreover, in order to further increase the inertia of the system about its longitudinal axis and to render the system aperiodic and practically nonsusceptible to disturbing influences of short duration, the 200-gram weight (see fig. 3 for details of upper part of suspension system) is furnished with two diametrically opposite vanes which dip into cups filled with a viscous

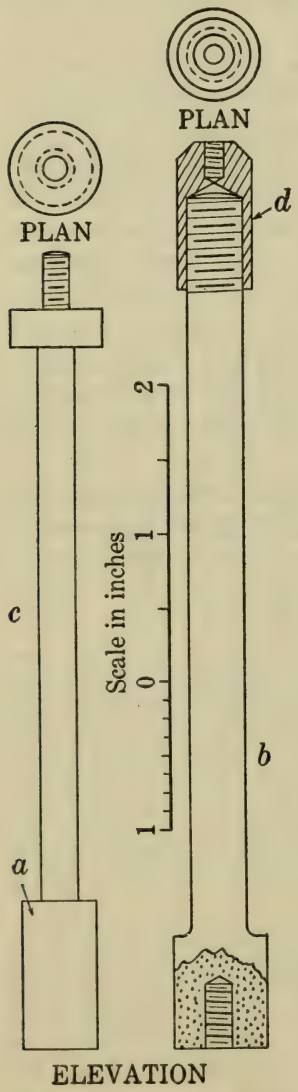

FIGURE 1.—Viscosity spindle and connecting rod. Left view, viscosity spindle—*a*, graphite spindle; *c*, spindle shaft. Right view, connecting rod—*b*, graphite rod; *d*, steel connecting collar.

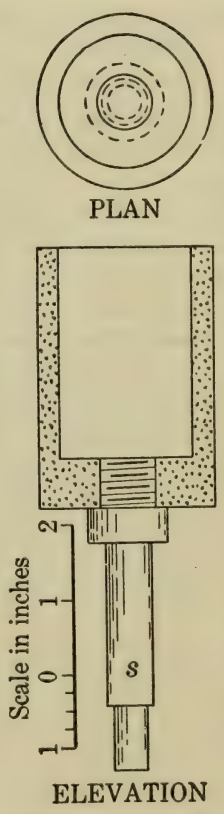

FIGURE 2.—Graphite crucible; *s*, supporting rod.

fluid, such as castor oil, at a distance of about 4 inches from the axis of the system.

The steel ribbon used was 0.15 millimeter thick, 1.65 millimeters wide, and 4.2 centimeters long, and was soldered at each end to brass

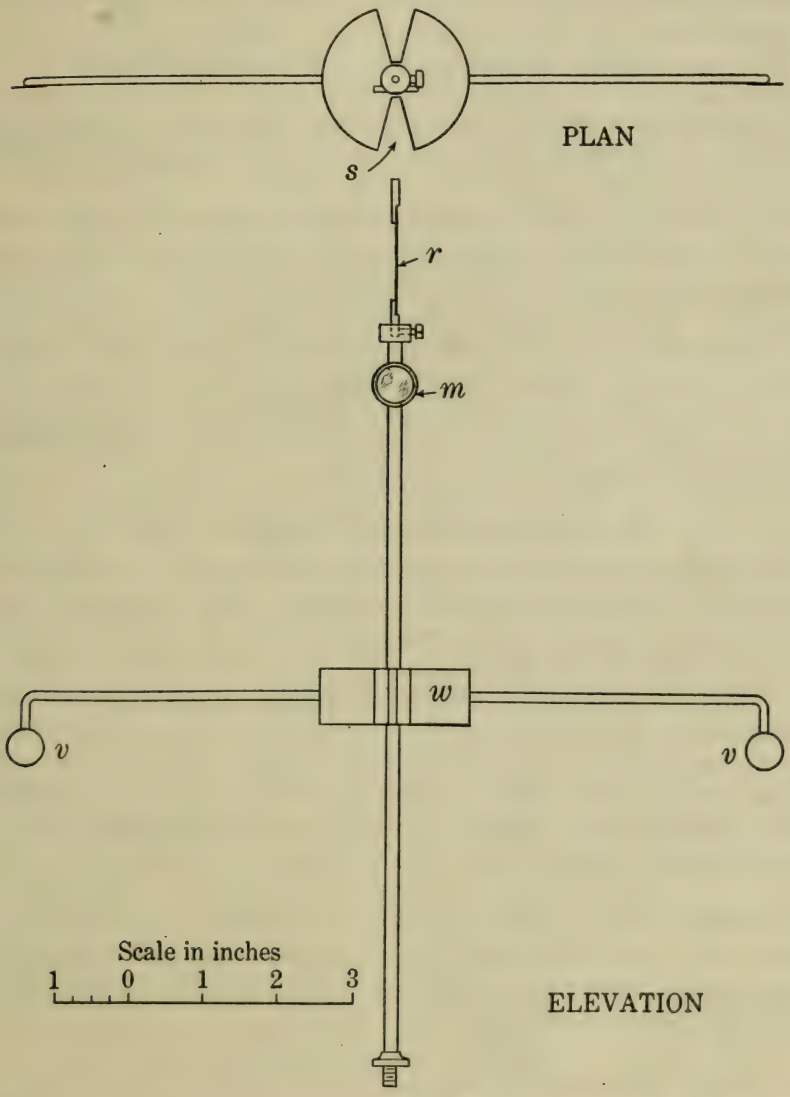

FIGURE 3.—Upper part of suspension system. *m*, mirror; *r*, steel suspension ribbon; *s*, open sector, *w*, weight; *vv*, vanes.

lugs which are held in position by small set screws. The maximum scale deflection, obtained in measurements made at the lower temperatures, corresponds to a total angular twist in the ribbon of about 7°. It is assumed that up to this point the displacement is propor-

tional to the torsion couple. The mirror deflection was measured in the usual way by means of a telescope and millimeter scale.

The entire suspension system is fixed to a rod that projects horizontally from an apparatus support firmly fixed to a heavy table and is provided with a millimeter scale for precise vertical adjustment of the apparatus. The furnace itself is first adjusted so that the steel shaft is in a vertical position, which insures the alignment of the crucible also. The point to which the suspension system is fixed is then aligned by means of a plumb bob with the axis of the shaft and crucible. In this manner the viscosity spindle is suspended centrally within the crucible.

The scale reading usually fluctuates slightly around a mean value, owing to unavoidable irregularities. As the fluctuation does not amount to more than one scale division, and the scale readings vary from about 20 to 200 millimeters, the maximum error introduced by this fluctuation is 0.5 to 5 per cent. At the lower scale readings, however, the measurement of temperature is subject to an experimental error of 10°, which will in general affect the correctness of the scale reading by about 10 per cent, as can be seen from the viscosity curves shown in figures 4 to 7 (pp. 21–24.)

THE MEASUREMENT OF TEMPERATURE.

The temperature of the slag was measured by means of an optical pyrometer of the Holborn-Kurlbaum type. The pyrometer lamp and absorption prism that was regularly used in the experiments had been calibrated by the Reichsanstalt. As a check on this lamp, it was compared at intervals with another lamp, which also had been calibrated by the Reichsanstalt, and was used only for comparison. The readings in all cases coincided within the allowable error of observation. As a further check on the lamp and the absorption prism, simultaneous readings of temperature were made with the optical pyrometer mentioned and a Bureau of Standards thermocouple (No. 443) in a furnace constructed in a manner similar to the well-known "black body" furnace of Lummer and Kurlbaum.[a] Without the absorption prism the corrections were less than 10° for temperatures ranging from 1,000° to 1,200° C. With the absorption prism the following corrections were noted:

Corrections for temperature readings.

Temperature.	Correction.	Temperature.	Correction.
°C.	°C.	°C.	°C.
1,200	+38	1,400	+43
1,250	+37	1,450	+44
1,300	+40	1,500	+50
1,350	+40		

a Lummer, O., and Kurlbaum, F., Der elektrisch geglühte "schwarze Körper": Ann. der Physik, vol. 5, 1901, p. 829.

The pyrometer is sighted directly upon the surface of the rotating slag through an open sector in the 200-gram weight. (See fig. 3.)

It is obvious that the rotation of the crucible containing the slag is instrumental in obtaining a uniform temperature throughout the slag mass, both on account of the shearing effect of the viscosity spindle, and the equalization of radiation from the heating tube.

OPERATION AND MANIPULATION OF APPARATUS.

The method of using the apparatus is as follows: 175 grams of slag is placed in the graphite crucible. This quantity of slag, when ground to 100 mesh, approximately fills the crucible when gently packed down in it. Although the height of the crucible internally is 7 centimeters the slag after having been melted and cooled forms a cylinder only 3.9 to 4.1 centimeters in length. The crucible, containing the slag, is properly placed, and the upper removable part of the furnace is fixed in position. The crucible is then rotated and the furnace is heated at such a rate that at the end of about 1½ hours the slag reaches a temperature of 1,200° C. The rate of heating is then decreased in order that the large mass of slag may acquire the temperature of the heating tube and diminish the lag effect. When the slag has become sufficiently fluid, as determined by several tentative lowerings of the viscosity spindle, the suspension system is carefully lowered until the viscosity spindle rests on the bottom of the crucible. During this operation the rotation of the crucible is stopped in order to avoid the possibility of breaking the graphite spindle while it is introduced into the viscous slag. The spindle is then raised exactly 0.5 centimeter above the bottom of the crucible, the adjustment being made by means of the scale in the apparatus support. If all types of slags possessed the same density at corresponding high temperatures the top of the viscosity spindle would be at a definite distance below the surface of the slag, and the end effect due to the immersed section of the spindle shaft would be constant for all types of slags.

Although different slags at a given high temperature undoubtedly have different densities it has not been considered necessary to determine exactly the length of the immersed part of the spindle shaft. It is probable that the greatest error that might be thus introduced is not more than 2 or 3 per cent; and in most cases is probably much less.

After the suspension system has been lowered and the depth of the viscosity spindle adjusted, the temperature of the slag is slowly raised at a rate of 2° to 5° C. per minute, the temperature and scale deflection being noted at approximately regular intervals. When the highest temperature desired, usually 1,500° to 1,600° C., is reached the heating current is so adjusted that the furnace cools at approxi-

mately the same rate that it was heated. Readings of deflection and temperature are taken, until the maximum deflection desired (corresponding to a viscosity of 2,000 to 3,000) is reached. The spindle is then raised out of the slag before the latter becomes hard enough to prevent withdrawal.

CALIBRATION OF THE VISCOSITY APPARATUS.

The viscosity apparatus is calibrated against pure castor oil whose viscosity at ordinary temperatures is known. This method of calibration has been generally used by previous investigators, particularly by Doelter and Arndt.

Kahlbaum and Raber [a] have found that the viscosity of castor oil in C. G. S. units (100 times as large as those referred to water at 20° C.) could be expressed for temperatures between 5° and 21° C. by the equation, $\eta = 59.301\ c^{-0.09040t}$. When t equals 20.5° C., η is equal to 9.297, or to 929.7 of the smaller units that are used exclusively hereafter in this report. Arndt [b] obtained measurements upon a sample of pure castor oil which agreed with those calculated by means of the above formula of Kahlbaum and Raber.

The sample of castor oil used by the author was apparently of high purity, was colorless, and as used was free from air bubbles. The following experimental results were obtained in a calibration at 20.5° C.

TABLE 1.—*Calibration data.*

[Distance of scale from mirror, 87 centimeters.]

Scale deflection.	Time of 1 revolution of crucible.	$K = \frac{(A \times B)}{87}$
Millimeters.	*Seconds.*	
57	1.90	1.25
62	1.77	1.26
73	1.45	1.21
85	1.24	1.21
105	1.04	1.25
110	.966	1.22
118	.890	1.21

A similar calibration with castor oil was made at 26° C. The viscosity calculated by means of a formula given by Kahlbaum and Raber for this temperature amounted to 394.4. The viscosity calculated from the calibration data obtained at 20.5° C. gave a value of 390.8. This agreement was considered satisfactory.

The temperature of the castor oil was in each case measured by means of a calibrated copper-constantan thermocouple and a Wolff potentiometer.

[a] Kahlbaum, —, and Raber, —, Acta. Ac. Leop., vol. 84, 1905, p. 204; see also Arndt, Kurt, Die Messung der Zahigkeit:Ztschr. Chem. Apparatenkunde, vol. 3, 1908, pp. 549–553.

[b] Arndt, K., Loc. cit.

REGULATION OF SPEED OF ROTATION.

The electric motor used in rotating the furnace shaft was operated by a 110-volt storage battery and showed a remarkably constant speed, which was controlled by means of a rheostat. The time required for at least 30 revolutions of the shaft should be taken by means of a stop watch; this was done a number of times during each experiment. The slow speed of the furnace shaft was obtained by means of suitable countershaft and pulleys between the motor and the shaft.

METHOD OF CALCULATING RESULTS.

In a determination of slag viscosity a record was made of the temperature of the slag, the scale deflection, the distance of the scale from the mirror, and the speed of rotation of the furnace shaft. The values of K, representing the scale deflection in millimeters at a distance of 1 centimeter from the mirror at a speed of rotation of 1 revolution per second, were calculated in a manner similar to that used in the calibration data of Table 1. For a spindle and suspension calibrated as described, the viscosity of the slag at various temperatures can be calculated by dividing the slag value of K by the observed castor-oil constant and multiplying the result by 929.7, the viscosity of castor oil at 20.5° C.

RESULTS OF VISCOSITY MEASUREMENTS.

The results of viscosity measurements on eight different slag samples (Lab. Nos. 22954, 22962, 22958, 22963, 24016, 23663, 22960, and 22968) are given in Table 2 for the temperature range 1,275° to 1,575° C. The composition of the slags is shown by the results of chemical analyses given in Table 3.

TABLE 2.—*The viscosity of typical blast-furnace slags at various temperatures.*

[H_2O at 20° C. = 1.]

Temperature, °C.	Viscosity of slag, Lab. No.—							
	22954	22962	22958	22963	24016	23663	22960	22968
1,275				1,400	2,600			
1,300			1,700	1,000	1,500		1,900	
1,325			1,060	820	1,080	2,000	1,100	3,000
1,350	2,600		800	680	800	740	840	1,800
1,375	1,150		640	540	620	560	640	1,220
1,400	750	3,000	510	460	500	480	510	1,000
1,425	540	580	420	400	420	410	440	820
1,450	410	480	350	350	350	360	380	680
1,475	320	415	300	310	290	300	320	580
1,500	230	380	260	280	250	250	280	480
1,525	160	340	225	250	230	200	240	400
1,550		290		220	205			340
1,575								310

TABLE 3.—*Results of analyses of the eight slags.*

Lab. No.	Composition of slag.												Total.
	SiO_2.	Al_2O_3.	Fe_2O_3.	FeO.	Fe.	TiO_2.	CaO.	MgO.	MnO.	CaS.	Alkalies.	Moisture.	
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
22954	18.30	35.30	0.00	0.47	0.05	0.58	31.24	9.69	0.35	4.07	0.50	0.02	100.57
22962	31.54	14.79	.00	.07	.22	.29	47.65	1.80	.21	3.56	.....	.10	100.23
22958	33.67	26.62	.00	.28	.51	.28	26.67	6.43	.33	4.86	.....	.12	99.77
22963	34.27	13.78	.00	.07	.28	.56	41.30	6.39	.55	3.35	.....	.04	100.59
24016	35.76	[a] 13.36	.....	.....	.....	.....	42.11	3.94	.49	3.70	.....	.....	99.36
23663	36.04	[a] 12.10	.....	.....	.....	.....	42.04	4.03	.35	3.92	.....	.....	98.48
22960	37.18	11.46	.00	.31	.11	.52	25.33	19.58	2.21	3.51	.....	.04	100.25
22968	43.56	9.48	.00	.21	.38	.19	40.18	2.08	.21	2.75	.50	.05	99.59

[a] Includes Fe_2O_3.

Temperature-viscosity curves were plotted, as shown in figures 4 to 7. The curves closely approximate in form a rectangular hyperbola. These curves contain all the experimentally determined points representing the temperature-viscosity relations observed in the heating and cooling of the slag sample. In this way any possible differences in the apparent temperature of the surface of the slag as measured by the optical pyrometer and the actual temperature within the slag mass are brought to light. On account of this fact, a smooth curve drawn through all the points must represent very closely the ideal conditions desired, the deviations tending to neutralize one another.

The temperature-fluidity curves for the eight slags mentioned are also given in figures 4 to 7. The values were calculated by means of the familiar relation, Fluidity = 1/Viscosity. As was to have been expected from the general shape of the viscosity curves, the curves representing the fluidity at various temperatures approximate in form a straight line.

The actual experimental data obtained in the case of slag No. 22958 are given in Table 4, which contains results obtained with rising and falling temperatures. Duplicate determinations on separate samples of slag gave results which agreed well within the known errors of measurement.

TABLE 4.—*Comparison of viscosity values obtained with rising and falling temperatures.*

[Results obtained in experiments with slag designated Lab. No. 22958.]

Rising temperature.		Falling temperature.[a]			
Temperature, °C.	Viscosity.	Temperature, °C.	Viscosity.	Temperature, °C.	Viscosity.
1,382	605	1,292	1,940	1,382	611
1,410	513	1,297	1,707	1,387	524
1,415	460	1,302	1,505	1,425	446
1,425	442	1,312	1,319	1,440	375
		1,322	1,074	1,466	323
1,463	320	1,337	922	1,470	304
		1,350	805	1,488	285
1,473	291	1,359	728	1,525	228
1,510	255	1,368	689		

[a] Results are given in reverse order, for easy comparison with results under "Rising temperature."

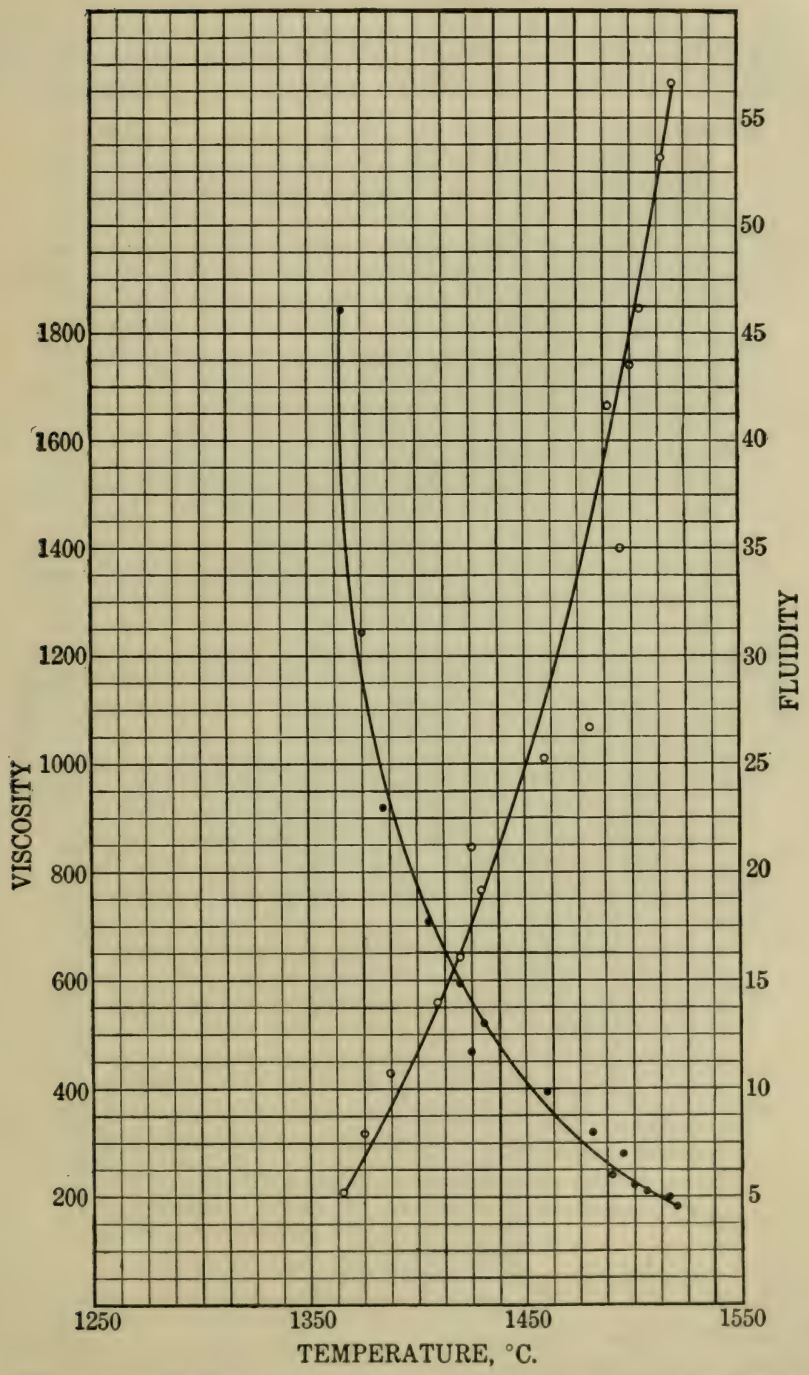

FIGURE 4.—Relations of temperature to fluidity and viscosity shown in experiments with slag No. 22954.

GENERAL APPEARANCE OF THE SLAG MELTS.

The rate of decrease in the temperature of the furnace on cooling was sufficiently slow to prevent the formation of amorphous glass

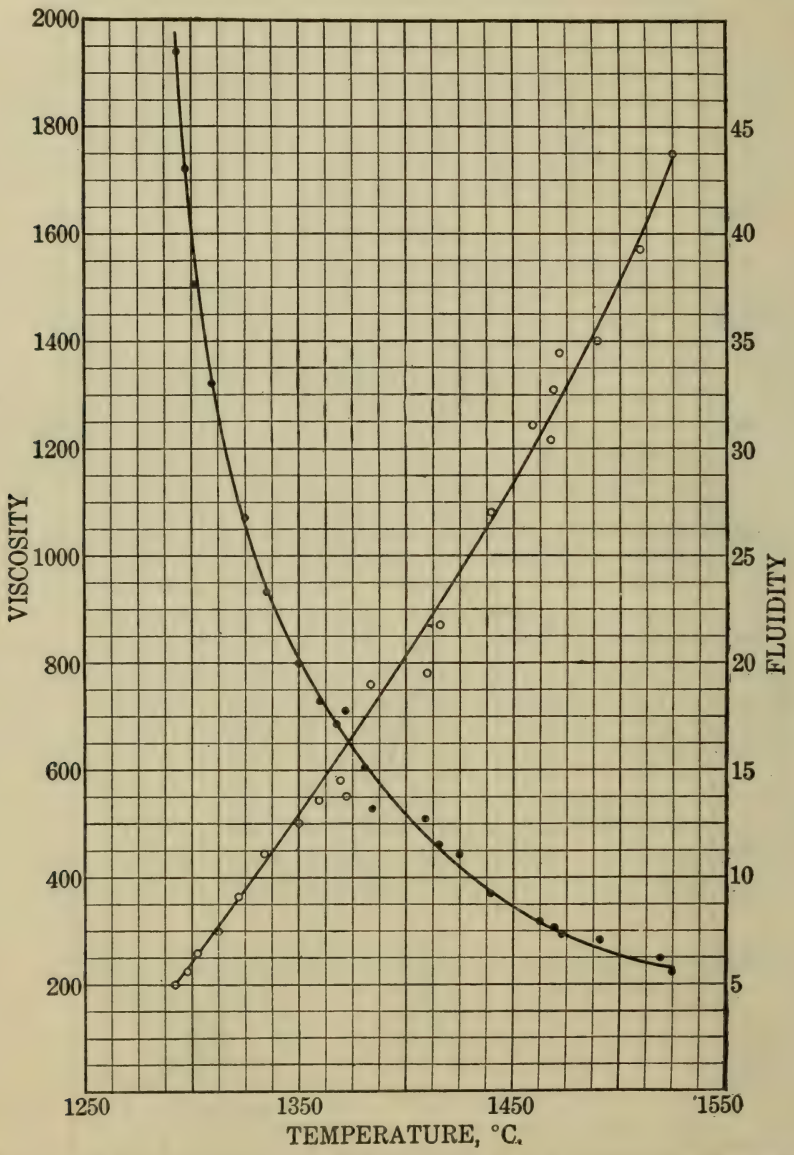

FIGURE 5.—Relations of temperature to fluidity and viscosity shown in experiments with slag No. 22958.

such as is frequently observed in slag samples cooled in an iron chill. The general physical appearance of the slag indicated that the conditions under which crystallization took place were uniform from the center of the melt to the outer surface, as far as could be judged by

examination under a microscope. There was no evidence of a glassy shell inclosing the melt. Crystals easily visible to the eye were frequently formed within the melt, but the crystalline structure of the slag was not studied systematically in connection with the experiments reported.

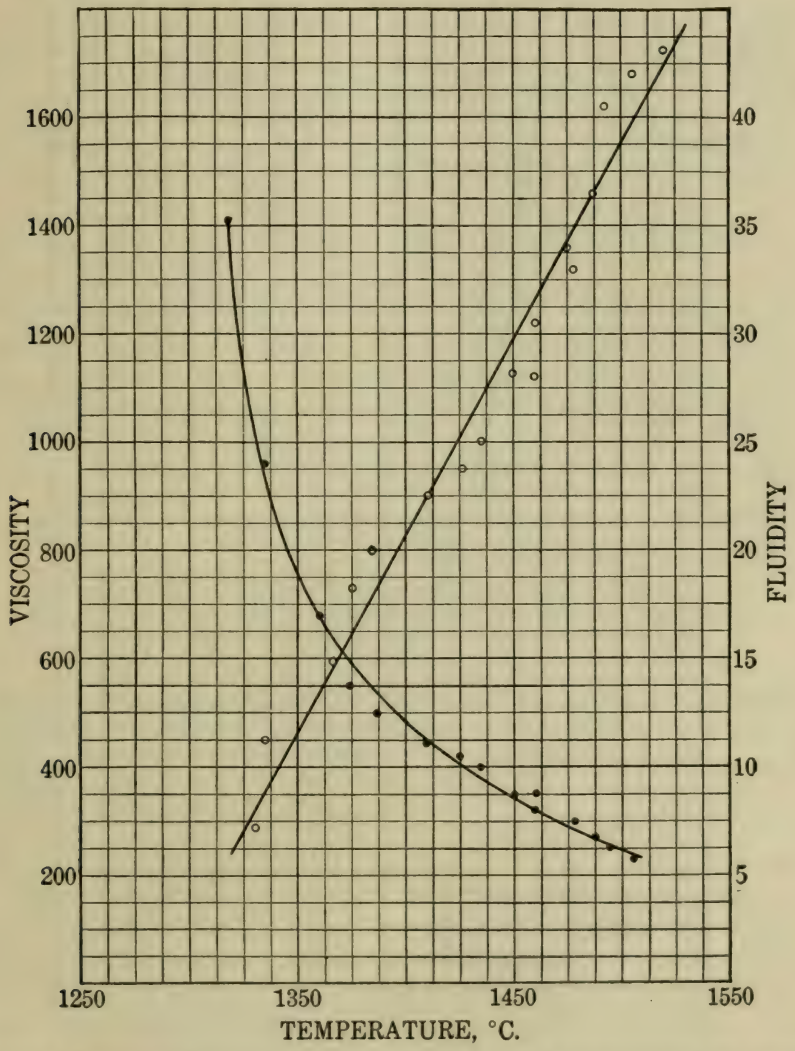

FIGURE 6.—Relations of temperature to fluidity and viscosity shown in experiments with slag No. 23663.

When cold the slag was easily removed from the graphite crucible by pressing on the bottom of the melt through the hole into which the graphite crucible support is threaded. The slag on cooling shrunk away from the graphite crucible wall sufficiently to prevent objectionable binding and sticking. The melt as taken from the crucible was cylindrical with an upper surface which is almost flat,

as the viscosity at the moment when the spindle was withdrawn was not great enough to prevent the slag from flowing into the space previously occupied by the graphite spindle.

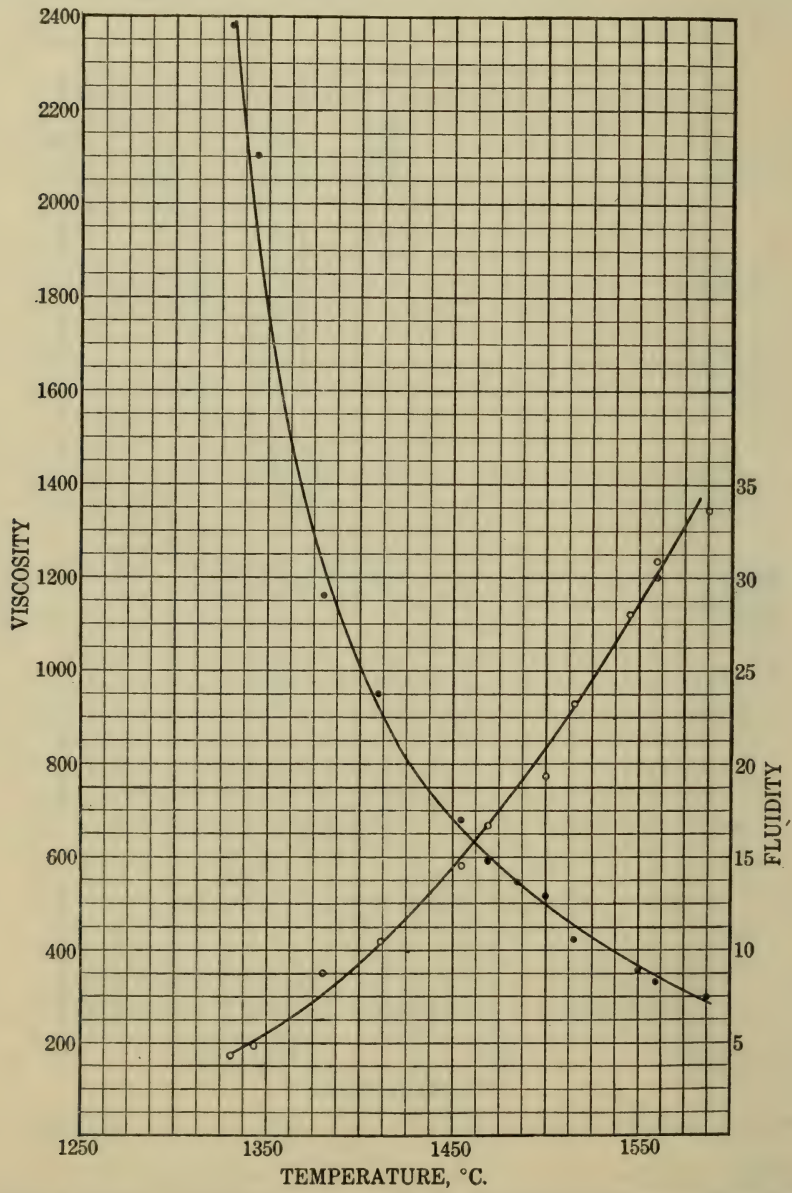

FIGURE 7.—Relations of temperature to fluidity and viscosity shown in experiments with slag No. 22968.

The separation of metallic iron, owing to the strongly reducing atmosphere of the furnace and the duration and intimacy of the contact between molten slag and the furnace atmosphere, was fre-

quently observed. These metallic globules, which in all probability closely approached the carbide in composition, were found mostly on or near the surface of the cooled melt; some were 2 to 3 millimeters in diameter.

DISCUSSION OF EXPERIMENTAL RESULTS.

If an average slag temperature of 1,500° C. is assumed, an examination of the data given in Table 2 discloses the fact that the viscosity of the normal blast-furnace slag as it flows from the cinder notch approximates 250 (H_2O at 20° C. = 1). A viscosity of about this value is what would be expected; it represents a viscosity less than that of castor oil at room temperature but greater than that of olive oil.

The viscosity value of the eight slags, considered in Table 2, at a temperature of 1,500° C. varied from 230 to 480.

If the maximum viscosity at which a slag acts satisfactorily in the furnace is assumed to be 300, it can be seen from Table 2 that the temperatures at which the various slags attain this viscosity vary over a comparatively large range. These temperatures are as follows:

TABLE 5.—*Temperature variation of slag at viscosity of 300.*

Lab. No. of slag.	Temperature, °C.	Lab. No. of slag.	Temperature, °C.
22954	1,480	24016	1,471
22962	1,545	23663	1,475
22958	1,475	22960	1,487
22963	1,483	22968	1,585

Ths slags designated Lab. No. 22962 and No. 22968 are decidedly more viscous than the rest, No. 22962 corresponding to a slag with high lime content and No. 22968 to a slag with high silica content. (See Table 3).

It would seem that even such high percentages of alumina as are present in the slags designated Lab. Nos. 22954 and 22958, although causing in the former a high initial softening temperature, do not have a very noticeable effect on the viscosity at higher temperatures. This peculiar property of alumina, which may be a quite general characteristic, is probably associated in some manner with the uncertainty of its action within the furnace.

COMPARISON OF VISCOSITY MEASUREMENTS WITH CONE TESTS.

It was thought a matter of interest to compare the results of viscosity measurements with cone tests made with corresponding slags. The test pieces were prepared according to a method which has been previously described.[a] The softening temperature tests were made,

[a] Fieldner, A. C., and Feild, A. L., A new method and furnace for the determination of the softening temperature of coal ash under fuel-bed conditions: Jour. Ind. and Eng. Chem., vol. 7, 1915, p. 829.

however, in a platinum wire resistance furnace in an atmosphere of air. For this reason the comparison is not a strict one because the state of oxidation of the iron content of the slag and of the sulphur content was not the same in the two cases. In the cone tests temperature measurements were made with a platinum and platinum-rhodium thermocouple, calibrated by the Bureau of Standards, and a calibrated Siemens-Halske millivoltmeter. The softening temperatures observed in cone tests made on six of the eight slag samples previously considered are given in Table 6. The softening temperature is expressed as an interval, the first temperature being the temperature of initial deformation and the last the "down" point.

TABLE 6.—*Softening temperatures determined by means of the cone method.*

Slag used (Lab. No.).	Softening temperature.	Calculated temperature at which slag attained a viscosity of 2,000.
	°C. °C.	°C.
22954	1,410 to 1,410	1,360
22962	1,403 to 1,443	1,410
22958	1,342 to 1,342	1,290
22963	1,343 to 1,360	1,265
22960	1,297 to 1,300	1,285
22968	1,279 to 1,279	1,340

In the third column of Table 6 are given the calculated temperatures at which the slag melts used in the viscosity determinations attained a viscosity of 2,000. The softening temperatures as determined by the cone test in air are higher than would be expected from the viscosity measurements. This may be partly due to the effect of the oxidizing atmosphere on the slag cone at elevated temperatures. In these tests, as was indicated by the viscosity measurements, the slag designated Lab. No. 22962 was found to be the most refractory.

SUMMARY.

A modification of the torsion method of Margules is applicable to the measurement of the viscosity of blast-furnace slag at high temperatures.

The upper limit of temperature, 1600° C., attained in the experiments is imposed by the furnace refractories, and not by inherent limitations in the viscosity apparatus.

A single run with the furnace gives a temperature-viscosity curve over a wide range of temperature.

Viscosity and fluidity curves are given for eight typical slags.

The temperature-viscosity curve in the cases given approximates in form that of the rectangular hyperbola, whereas the temperature-fluidity curve approaches a straight line.

The average viscosity at 1500° C. of eight slags was found to be 301 (H_2O at 20° C.=1), corresponding to a viscosity less than that of castor oil at room temperature but greater than that of olive oil.

A refractory slag—that is, a slag possessing a high initial softening temperature—is not necessarily more viscous at high temperatures than a more fusible slag. This fact was shown by experiments with a slag containing 35.3 per cent alumina.

It is thought that the method described for measuring the viscosity of blast-furnace slags at high temperature is applicable, with suitable modifications, over a much wider range of viscosity than was considered necessary in these experiments.

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